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वस्त्र रंजक सामग्री — रंगाई विधि द्वारा क्रोम रंजकों की तीव्रता एवं रंग के मूल्यांकन की विधि

(पहला पुनरीक्षण)

Textile Dyestuffs — Method for Evaluating Strength and Shade of Chrome Dyes by Dyeing Test

(First Revision)

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

The method laid down in this standard for determining the strength of dyestuffs against a mutually accepted standard would be useful for assessing both the strength and the shade of the dyestuff. The method prescribed in the standard is a general method and wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings of the dyestuffs. It is generally better to use medium shades (about 1 percent), as they show any variations in strength more clearly than dark shades. Wherever necessary, it is advisable to carry out evaluation in deep shades also as recommended by the manufacturers (for example, blacks, navy blue, etc).

Chrome dyes are generally dyed by the following three methods:

- *After chrome process (or top chrome process)* In this process, dyeing is first carried out on the wool and subsequently chroming is done in the same bath.
- ii) One bath chrome process (chromal process or chromate process or metachrome process)In this process, dyeing and chroming take place simultaneously.
- *Bottom* chrome *process* (*chrome mordant process*) In this process, chrome mordant is first fixed on the wool and subsequently dyed in a fresh bath.

The method prescribed in this standard is based on 'After chrome process' as it is applicable to wide range of dyes. However, an outline of other two methods of dyeing are given in Annex B which may be used if agreed to between the buyer and the seller.

This standard was first published in 1975. The first revision has been made in the light of experience gained since its publication and to incorporate the following major changes:

- a) Title of the standard has been modified;
- b) Grade and purity of chemicals used have been specified;
- c) Sampling clause has been modified; and
- d) References to Indian Standard have been updated.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

TEXTILE DYESTUFFS — METHOD FOR EVALUATING STRENGTH AND SHADE OF CHROME DYES BY DYEING TEST

(First Revision)

1 SCOPE

This standard prescribes a method for evaluating strength and shade of chrome dyes by dyeing test. This standard does not include automated instrumental method.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

Title
Specification for sodium sulphate, anhydrous (Technical grade)
•
(second revision)
Acetic acid – Specification (fourth
revision)
Reagent grade water –
Specification (third revision)

3 SAMPLING

- **3.1 Lot** All the containers of the same dye and the same strength delivered to a buyer against a dispatch note shall constitute a lot.
- **3.2** Unless otherwise agreed to between the buyer and the seller the number of containers to be selected at random from a lot shall be in accordance

with Table 1.

3.3 From each container draw small quantities of the dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

Table 1 Sample Size (*Clause* 3.2)

Lot Size	Sample Size
(1)	(2)
2 to 15	2
16 to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 and above	8

4 STANDARD DYESTUFF

The standard sample of dyestuff against which the strength of dyestuff under test is evaluated shall be as agreed to between the buyer and the seller.

5 QUALITY OF REAGENTS

Unless otherwise specified analytical reagent grade chemicals with 99.0 percent purity shall be employed in tests and distilled water (*see* IS 1070) shall be used where the use of water as reagent is intended.

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6 EVALUATION OF STRENGTH OF DYESTUFF

6.1 Preliminary Test to Determine Strength

Before dyeing the hanks, carry out a preliminary test to determine the approximate strength of the dyestuff under test as given in **6.1.1**.

6.1.1 Take separately 10 ml of 0.1 percent solutions of the dyestuff under test and the standard dyestuff (*see* **A-4.1**). Put a spot or streak or pour each of the solutions of the dyestuff under test and the standard dyestuff on filter paper. Visually, examine the spots. If they are not equal in strength, dilute the stronger solution with water to such an extent as to get approximate equal strength of dyestuff spotted on filter paper. Calculate the approximate strength of the dyestuff under test by the following formula:

Approximate strength (Sa) of dyestuff under test =

$$\frac{Vt \times 100}{Vs}$$

Where

Vt = final volume in millilitres of the solution of dyestuff under test, and

Vs = final volume in millilitres ~of the solution of the standard dyestuff.

- **6.2** Prepare solutions of standard dyestuff and dyestuff under test as given in **A-4.1**.
- **6.3** Prepare dyeings of 0.95, 1.00 and 1.05 percent depths of shade of the standard dyestuff as given in **A-4.2.1**.
- **6.4** Similarly, prepare dyeings of 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade of dyestuff under test as given in **A-4.2.2**.
- **6.5** Carry out the dyeings of the fresh hanks in the exhaust liquors of the above dyeings by following the procedure given in **A-4.3**.

6.6 Assessment

Compare the dyeings of dyestuff under test (see

6.4) and standard dyestuff (*see* **6.3**) in north skylight (*see* Note). Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentages of the dyeings which match exactly.

NOTE — Before comparing the dyeings, they shall br spread out properly. The dried hanks shall be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of backing on the appearance. The hanks shall be compared, if possible, in north skylight. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test shall be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings shall be repeated. If the dyeings of the test sample and the standard do not fall within range, fresh set of dyeings shall be taken.

6.7 For Deep Shades

Wherever necessary, evaluation shall also be carried out in deep shades on similar lines as for one percent shade, using dyeing assistants in accordance with the manufacturers' recommendations.

7 CALCULATION

Calculate the strength of the dyestuff under test by the following formula:

$$S = \underline{A} \times S_a$$

Where

S = strength of dyestuff in percent,

A = percentage dyeing of the standard dyestuff, and

B = percentage dyeing of the dyestuff under test which matches with A.

 S_a = approximate strength of the dyestuff under test as determined under **6.1.1**.

8 REPORT

The report shall include the following information:

a) The strength of dyestuff in percent (see 7),

- b) Shade of the dyestuff in comparison with that of the standard dyestuff, and
- c) Shade of the exhaust dyeings in comparison with that of the exhaust dyeing of the standard dyestuff.

ANNEX A

(Clauses 6.1.1, 6.2, 6.3, 6.4 and 6.5)

GENERAL METHOD FOR DYEING OF CHROME DYES ON WOOL

A-1 APPARATUS

A-1.1 Dye Vessels — porcelain or stainless steel dye vessels.

A-1.2 Graduated Pipettes — capable of measuring correct to 0.1 ml.

A-2 DYEING ASSISTANTS

A- 2.1 Water — Distilled water (*see* IS 1070) shall be used in the preparation of the dye-bath.

NOTE — For rinsing, water hardness of not more than 50 ppm expressed as calcium carbonate may be used.

A-2.2 Sodium Sulphate (Calcined) Solution — 20 percent (m/v) (see IS 255).

NOTE — Quality of sodium sulphate solution is extremely important. Commercial brands may be acidic or alkaline, therefore they shall be neutralized.

- **A-2.3 Acetic Acid Solution** 10 percent (m/v) (see IS 695).
- **A-2.4 Formic Acid Solution** 10 percent (m/v).
- **A-2.5 Potassium or Sodium Dichromate Solution** 5 percent (m/v).
- **A-2.6 Ammonium Sulphate Solution** 10 percent (m/v).
- **A-2.7 Ammonium Acetate Solution** 10 percent (m/v).

A-3 PREPARATION OF THE HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, 100 percent pure wool knitting yarn having no finishing

chemical, blueing agent or fluorescent brightening agent shall be used. Each hank shall weigh 5 ± 0.1 g (*see* NOTE).

NOTE — If the mass of the hank is not 5 ± 0.1 g then it shall be weighed accurately and the amount of dyestuff and the chemicals to be taken shall be calculated accurately.

A-3.2 Preparation of the Test Hanks

Treat the required number of hanks in warm water (not higher than 60°C) for 10 minutes, squeeze evenly to contain approximately its own mass of water, cool and enter into the dye-bath.

A-4 PROCEDURE

A-4.1 Preparation of the Dyestuff Solutions

- **A-4.1.1** Weigh accurately 1.0 g of the dyestuff under test. Paste the dyestuff thoroughly with cold water and dissolve by adding hot water. If necessary, heat the solution till it becomes clear. Dilute the solution with cold water and make up 1000 ml in a volumetric flask.
- **A-4.1.2** Similarly, weigh accurately $(1.00 \times 100 \text{ g})/$ Sa of dyestuff under test (see **6.1.1**) and prepare solution following the procedure given in **A-4.1.1**.
- **A-4.2** Dyeing (for 5 ± 0.1 g Hank)
- **A-4.2.1** Pipette out separately 47.5, 50.0 and 52.5 ml of standard dyestuff solution in the dye vessels so as to give 0.95, 1.00 and 1.05 percent depths of shade. Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the final volume to 300 ml (liquor to material ratio 60:1). Add 2.5 ml of sodium sulphate (calcined) solution and 0.5 ml acetic acid solution. Stir the dye liquor and raise the temperature to 60°C. Enter the wetted hanks and turn them frequently so as to obtain level dyeings. Work for 10 minutes at this

temperature. Remove the hank from the dye-bath, add further 0.5 ml of acetic acid solution and stir well. Enter the hanks into the dye-bath and turn the hanks frequently. Slowly raise the temperature of water-bath to boil within 20 minutes. Continue the dyeing at boil for 30 minutes (see Note). Remove the hanks from the dye-bath and add 0.5 ml formic acid solution enter the hanks, and boil further for 30 minutes. Then cool down the water-bath to 85°C and add 0.5 ml of sodium or potassium dichromate solution. Raise the temperature to boil and continue the dyeing further for 45 minutes. At the end of dyeing squeeze the dyed hank, returning the squeezed out solutions to the respective dye-baths. Then rinse the dyed yarn with warm water (50 to 60°C). Dry the hanks in oven or in drier at a temperature not exceeding 70°C.

NOTE — During dyeing, water should be added to make up the volume lost by evaporation.

A-4.2.2 Similarly, pipette out separately 45.0, 47.5, 50.0, 52.5 and 55.0 ml of solution of dyestuff under test in the dye vessels so as to give 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade (in terms of equivalent standard strength). Dye the hanks by following the method given in **A-4.2.1** (*see* Note).

NOTE — The dyeings with the solutions of the dyestuffs under test and of the standard dyestuff should be done simultaneously in the same water-bath.

A-4.3 Exhaust Dyeing — Make up the volumes of the exhaust liquor (*see* **A-4.2.1** and **A-4.2.2**) to the original volume (300 ml). Enter the fresh wetted hanks in the exhausted dye-baths and continue the dyeing for 30 minutes, at boil. Squeeze the hanks, rinse in warm water (50 to 60°C) and dry.

ANNEX B

(Foreword)

CHROME PROCESSES

B-1 ONE-BATH CHROME PROCESS

B-1.1 Pipette out separately the required amount of standard dyestuff solution (see A-4.2.1) to get different depths of shade. Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the final volume to 300 ml. Add requisite quantities of sodium sulphate solution, 2 ml sodium or potassium dichromate and 3 ml ammonium sulphate or ammonium acetate solution. Stir the dye liquor and raise the temperature to 60°C. Enter the wetted hanks and turn them frequently so as to obtain level dyeings. Work for 10 minutes at this temperature and slowly raise the temperature of water-bath to boil within 30 minutes. Continue the dyeing at boil for 60 minutes. At the end of dyeing, squeeze the dyed hank returning the squeezed out solution to the respective dye-baths. Then rinse the dyed yarn with warm water (50 to 60°C). Dry the hanks in oven or in drier at the temperature not exceeding 70°C.

B-1.2 Similarly, pipette out separately required amount of solution of the dyestuff under test in different dye vessels (*see* **A-4.2.2**) to get different depths of shade and carry out dyeing as in **B-1.1**.

B-2 BOTTOM CHROME PROCESS

B-2.1 Chroming-Prepare chroming bath using 1.5 ml sodium or potassium dichromate and 0.70 ml formic acid solution and finally make the volume

300 ml. Stir the liquor of the bath and raise the temperature to 50°C. Enter the wetted hanks and turn them frequently so as to obtain uniform chroming. Slowly raise the temperature to boil within 20 minutes and continue the chroming at boil for further 60 minutes. At the end of chroming period, squeeze the hanks and rinse the same in cold water. Carry out the dyeing immediately.

B-2.2 Dyeing

Pipette out separately the required amount of standard dyestuff solution in the dye vessels to get different depths of shade (*see* **A-4.1.1**). Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the volume to 300 ml. Add 0.6 ml acetic acid solution, stir the dye liquor and raise the temperature to 40°C. Enter the chromed hanks and turn them frequently so as to obtain level dyeing. Slowly raise the temperature of water-bath to boil within 30 minutes. Continue the dyeing at boil for 90 minutes. At the end of dyeing, squeeze the dyed hanks and rinse the hanks with warm water (50 to 60°C). Dry the hanks in oven or in drier at the temperature not exceeding 70°C.

B-2.3 Similarly, pipette out separately required amount of solution of the dyestuff under test (*see* **A-4.1.2**) in different dye vessels to get different depths of shade and carry out chroming and dyeing as in **B-2.1** and **B-2.2**.

ANNEX C (Foreword)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee, $\,$ TXD 07

Organization	Representative(s)
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